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Crystal diffraction and photo electron measurements of *K* and *L* levels in the elements 47 Ag to 52 Te

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With 2 figures in the text

ABSTRACT

Precision measurements by crystal diffraction of the $K\alpha_1$, α_2 X-ray emission lines in the elements 47 Ag to 52 Te, with an accuracy of ± 0.004 XU or ± 0.3 eV, are reported. The L_{I} and L_{II} levels in the same elements are determined with the photo electron method with an accuracy of ± 0.4 eV. Together with previous photo electron data on the L_{III} levels, and by addition of the K emission line energies to obtain the K levels, all of the four deepest atomic K and L level energies are given within ± 0.5 eV.

1. Introduction

Precise knowledge of atomic binding energies has beomce important not only in atomic and solid state physics but also in nuclear spectroscopy. The precision photo electron method, developed by Siegbahn, Nordling, and Sokolowski [1] for measurements of the binding energies with an accuracy of a few tenths of an electron volt, has been employed for studies of *K*, *L*, *M*, and *N* levels in the fourth to the seventh periods of the periodic table [2–7]. In the present work, this technique has been applied to L_{I} and L_{II} levels in the elements 47 Ag to 52 Te, where the L_{III} levels have been previously established by one of us (C. N.) [5].

For a determination of the *K* levels of these elements, experimental limitations render the application of the photo electron method difficult. We must therefore resort to *K* X-ray emission line measurements of comparable absolute accuracy. These have been carried out in direct emission by one of us (P. B,), employing the precision curved crystal diffraction spectrometer described in previous reports [8–11]. The *K* levels are obtained by addition of the $K\alpha_1$ and α_2 lines to the L_{III} and L_{II} levels respectively. At the same time two independent determinations of the L_{II}–L_{III} level differences are obtained, providing a check on the consistency of the measurements [7].

2.1 X-ray measurements

Precision measurements of *K* emission spectra of the elements 47 Ag to 52 Te have previously been made by various authors using the Siegbahn "tube" spectrometer [16, 17, 19, 20, 21], by Bearden with the aid of the two-crystal spectrometer [13], and by Ingelstam with a curved crystal arrangement [18]. The registration has, except the work of Bearden, been photographic. The present measurements introduce the improvement on the precision which is implied by electronic counting and recording of line profiles. The spectrometer design introduces several additional advantages. The registration of a spectral line in the two mirror-symmetric positions of the spectrometer, and the subsequent determination of Bragg angle through a superposition of the profiles, eliminates systematic errors caused by minute spectral window asymmetries. The observed line profiles appear symmetric within experimental error, which is expected since the diffraction pattern in the Laue (transmission) case is symmetric. The superposition of the two lines need not be carried out literally, the important point being that the line peak positions are established identically for both profiles. The mechanical advantages of the spectrometer construction, which involves no requirements of precision motion other than the simple rotation of the crystal around a vertical axis, have been discussed previously [8]. The fact that errors, arising from aberration and adjustment uncertainties, are to a large extent linear with wavelength, and therefore automatically corrected for by the calibration procedure, has also been elaborated in the previous report.

For the calibration only one reference line is necessary, and as such one of the most well-established X-ray lines, Mo $K\alpha_1$, has been chosen. The wavelength for this line has been measured with high precision, notably by Larsson [12] and Bearden [13], relative to the atomic spacing in calcite, which constitutes the Siegbahn definition of the X unit. In practise, the wavelength value according to Larsson, 707.831 XU, has long been used as a wavelength standard, a fact which has caused Du Mond [14] to propose its use as a definition of the X unit. The Bragg angle for this line employing quartz $(13\bar{4}0)$ is the largest one measurable with the present spectrometer, and consequently the calibration error is always a small, sometimes negligible contribution (6 ppm) to the total uncertainty in the wavelength measurements.

The resolution of the spectrometer for 0.1 mm slit width varies between 0.03 % and 0.04 % from Ag $K\alpha_2$ to Te $K\alpha_1$ [9]. The contribution of natural line widths is not included in these figures. The high counting rates and signal to background ratios obtained are illustrated in Fig. 1 for the silver $K\alpha$ lines. The recordings for the other elements are closeley similar, although Fig. 1 represents the most unfavourable element on account of the increasing absorption at low energies. In fact, the wavelength region of this investigation is the instrument's most favourable one, enabling determinations with a relative accuracy of better than $1:10^5$.

The experimental procedure has been described in previous reports [8], [10]. The targets in the X-ray tube consisted of copper discs, 1.5 mm thick, with the pure metals soldered in 0.5 mm thick layers on top, except for silver, which was used in the form of a pure metal disc, 2 mm thick. The focal spot of the electron beam left little or no traces on the metallic surfaces. It should be pointed out that due to the high energy of the impinging electrons, 500 keV, most of the X rays come from the deeper layers of the targets, so that the state of the surface during the operation becomes unimportant. The heat dissipation is 20 watts, and the target is easily cooled.

In view of the low energies of the spectral lines measured in this work, the possibility of systematic errors, due to attenuation of the line profiles by differential absorption, will be discussed briefly. Absorption of the X-ray beam takes place in the target, in various windows, in the air path, and in the 1.5 mm thick quartz crystal. Calculation of the total absorption, using the tables by Davisson [15], yields a transmitted to incident ratio of 3 % for silver $K\alpha$ radiation. The situation

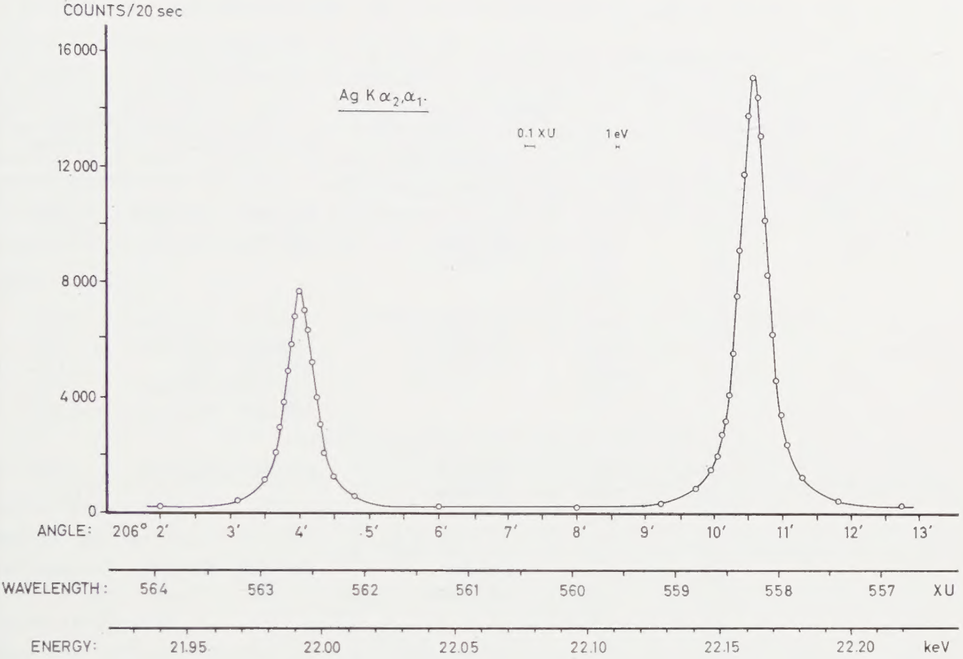

Fig. 1. A typical recording from the present measurements of the $K\alpha_1$, α_2 doublet line. The resolution is here 0.03 %.

is even worse for the calibration line, Mo $K\alpha_1$, which is attenuated to 0.06 % of the primary intensity. These figures agree with estimates from the observed reduction of line intensities. Further calculation of the variation in attenuation with energy over a line profile yields a change of 0.7 % for Mo $K\alpha_1$ as we pass the natural line width for this radiation. This would not be observable as a line deformation, and the influence on peak position is negligible. The effect is even smaller for lines of higher energy.

2.2 *Experimental results*

The calibration of the spectrometer with the aid of Mo $K\alpha_1$ was carried out immediately before, and was checked after the set of measurements. The result is reported in ref. [10]. Four Bragg angle measurements, each consisting of the delineation of two profiles were made for each of the two $K\alpha$ lines of the six elements, the results reported in Table 1. The mean values of Bragg angles (corrected to a standard temperature of 18°C), wavelengths in XU, and energies in keV are given in the fourth column in Table 1, and the previous measurements appear in column 5.

2.3 *Error estimate*

According to the accumulated experience from measurements with the present instrument (comprising about 350 profile delineations) the line peaks are established with a standard deviation of 1/80 of the line width at half maximum. The double standard deviation is chosen as an error limit ("maximal error"), corresponding to

Table 1. Present and previous X-ray measurements.

(Quantum conversion factor: 12 372.44 keV. XU [23].)

Line	Bragg angles 18°C	t °C	Mean Bragg angle **Wavelength, XU** Energy, keV	Previous measurements, Wavelength, XU, and author
Ag $K\alpha_2$	13° 49′ 15.7″	22.6	13° 49′ 15.9″	562.669 Kellström [16]
	16.1	23.1	**562.623**	562.638 Elg [17]
	15.9	23.1	21.9906	
	16.0	23.0		
Ag $K\alpha_1$	13° 42′ 41.4″	23.2	13° 42′ 41.2″	558.241 Ingelstam [18]
	41.3	23.0	**558.246**	558.255 Bearden [13]
	40.9	23.0	22.1631	558.235 Elg [17]
	41.0	23.0		558.252 Nilsson [19]
				558.243 Beckman et al [8]
Cd $K\alpha_2$	13° 12′ 45.2″	21.3	13° 12′ 45.2″	
	45.4	21.4	**538.303**	538.317 Valasek [20]
	45.4	21.3	22.9842	
	44.8	21.2		
Cd $K\alpha_1$	13° 6′ 9.5″	18.2	13° 6′ 9.5″	
	9.4	18.6	**533.903**	533.904 Valasek [20]
	9.6	20.9	23.1736	
	9.5	20.8		
In $K\alpha_2$	12° 38′ 33.7″	23.2	12° 38′ 33.8″	
	33.7	23.1	**515.474**	515.484 Valasek [20]
	34.0	23.5	24.0021	
	33.8	22.3		
In $K\alpha_1$	12° 31′ 56.8″	22.7	12° 31′ 56.8″	
	56.5	22.0	**511.050**	511.064 Valasek [20]
	57.0	22.4	24.2099	
	56.8	21.8		
Sn $K\alpha_2$	12° 6′ 30.6″	22.1	12° 6′ 30.7″	
	31.0	22.5	**494.026**	494.016 Stenman [21]
	30.8	22.9	25.0441	
	30.5	22.5		
Sn $K\alpha_1$	11° 59′ 52.8″	21.9	11° 59′ 52.9″	
	53.0	23.7	**489.584**	489.572 Stenman [21]
	52.9	23.0	25.2713	
	52.8	22.6		
Sb $K\alpha_2$	11° 36′ 24.2″	23.4	11° 36′ 24.5″	
	25.0	23.3	**473.843**	473.834 Ingelstam [18]
	24.3	23.6	26.1109	
	24.6	23.6		
Sb $K\alpha_1$	11° 29′ 45.3″	22.5	11° 29′ 45.5″	469.374 Ingelstam [18]
	45.4	22.5	**469.379**	469.372 Nilsson [19]
	45.6	22.8	26.3592	**469.373** Beckman et al [8]
	45.8	23.0		
Te $K\alpha_2$	11° 8′ 7.3″	23.1	11° 8′ 7.2″	
	7.0	23.3	**454.843**	454.832 Ingelstam [18]
	7.0	23.2	27.2016	
	7.3	23.1		
Te $K\alpha_1$	11° 1′ 27.5″	22.9	11° 1′ 27.3″	
	27.2	22.8	**450.362**	450.353 Ingelstam [18]
	27.1	22.4	27.4722	
	27.4	22.3		

a confidence coefficient of 95 %. The error of the mean of four measurements of Bragg angle is obtained by dividing by $\sqrt{8}$ [8]. For the present set of mean Bragg angles we thus get 0.25″ (seconds of arc) as a measure of accidental errors. Allowance for non-linear systematic errors, mainly in the angular scale divisions, is made by assigning an uncertainty of 0.2″ for each profile [8], which constitutes 0.15″ in the Bragg angle. Quadratic addition gives a total error of 0.3″ for all the Bragg angles in this work. By inclusion of the calibration uncertainty, 6 ppm [10], wavelength errors of 0.0048 XU (Ag) to 0.0044 XU (Te) and energy errors of 0.19 eV (Ag) to 0.27 eV (Te) result. These are rounded off to the following values:

Error limits:

	Bragg angle	Wavelength, XU	Energy, keV
Ag, Cd, In:	±0.3″	±0.005	±0.2
Sn, Sb, Te:	±0.3″	±0.005	±0.3

2.4 Comparison with previous measurements

It should be noted that two of the present X-ray lines, Ag $K\alpha_1$ and Sb $K\alpha_1$, were measured about one year before the present investigation with the same spectrometer, employing a slightly different part of the precision scale, and in connection with a separate calibration with Mo $K\alpha_1$. The calibration constant is to a minute extent dependent on the exact geometrical alignment of the crystal. Due to a complete readjustment of the spectrometer, undertaken before the present investigation, the two calibrations showed a difference of 30 ppm. The wavelength values obtained relative to Mo $K\alpha_1$ on the two occasions showed, however, an agreement well within error limits. The two wavelength values of Ag $K\alpha_1$ and Sb $K\alpha_1$ can be calculated from Bragg angles given on page 434 of ref. [8]. Unfortunately, computational errors in the temperature corrections have yielded slightly erroneous values for the mean Bragg angles. As can be verified from the uncorrected angles and temperatures given in the text of ref. [8], the mean of the Bragg angles should be increased by 0.3″ for Sb, 0.1″ for Ag, and 0.2″ for Mo $K\alpha_1$. Using the correct figures, the wavelengths for Ag $K\alpha_1$ and Sb $K\alpha_1$ cited in this paper, in column 5 of Table 1 (Beckman *et al.*) are obtained, in good agreement with the present data, which gives strong support to the reproducibility of the measurements with the spectrometer.

Both the above mentioned lines have been measured previously by more than one investigator. The several Ag $K\alpha_1$ determinations have been discussed by Ingelstam [18], Sandström [22], and Beckman *et al.* [8]. The wavelength reported here is seen to reconcile the low values by Elg [17] and Ingelstam [18] with the high values found by Bearden [13] and Nilsson [19]. The Sb $K\alpha_1$ line has been measured by Ingelstam [18] and Nilsson [19], in good agreement with the result obtained with this spectrometer.

As can be seen from a comparison of columns 4 and 5 in Table 1, the agreement between present and previous data is within 0.015 XU, as expected from the error limits pertaining to column 5. Only two of the previously accepted values, however, fall within the present error limits, 0.005 XU, of the figures in column 4, six values fall below and four above.

A further check on the accuracy of the present measurements is obtained by comparing the $K\alpha_1$–α_2 doublet splittings with the L_{II}–L_{III} level energy differences obtained by the photo electron method. A comparison can also be made with values of the spin doublet splitting according to L emission spectra. This is seen in Table 4.

3.1 Photo electron measurements

Measurements of L level energies in fifth period elements, using the photo electron method have been reported by one of us (C. N.) in ref. [5]. In this reference the L_{III} energy only was determined for the majority of elements, the exceptions being Ag and Sn, for which the L_{II} and L_{I}, L_{II} energies respectively were also measured. All L_{III} energies were determined with the $^1D_2$ KLL Auger line in copper as a reference line. The predominant part of the errors assigned to the energy values was due to the uncertainty in the momentum of this calibration line. In the present investigation the measurements have been extended to the L_I and L_{II} levels, but here the L_{III} (Cu$K\alpha_1$) photo line of each element has been used for calibration purpose. The momentum values of these photo lines (Table 2, column 1), have been taken from ref. [5]. The error involved in this procedure is insignificantly larger than that of a direct calibration with the copper Auger line, and from the experimental point of view great simplicity is achieved, since no triple source has to be made.

Four measurements were made of the L_I and L_{II} energies in each of the elements Ag, Cd, In, Sb and Te, the photo electron energies ranging from 4.5 keV for the AgL_{II}(Cu$K\alpha_1$) line down to 3.1 keV for the TeL_I(Cu$K\alpha_1$) line. The Cu$K\alpha_1$ X-radiation was used throughout the measurements for the production of photo electrons. At least two sources were prepared of each element as can been seen in the second column of Table 2, and the L_I, L_{II} and L_{III} photo lines were always recorded in the

Table 2. Peak current values of photo electron lines.

Element Calibration line Momentum (gauss-cm) [5]	#	I_{L_I} (amp)	$I_{L_{II}}$ (amp)	I_{cal} (amp)
Ag	177	2.88358	2.97851	3.03532
AgL_{III} (Cu$K\alpha_1$)	178	2.88296	2.97795	3.03455
$\mathrm{B}\varrho_{cal} = 231.516$	190	2.88318	2.97818	3.03476
	198	2.88108	2.97601	3.03278
Cd	182	2.81215	2.91243	2.97613
CdL_{III} (Cu$K\alpha_1$)	182	2.81221	2.91240	2.97594
$\mathrm{B}\varrho_{cal} = 226.846$	190	2.81150	2.91176	2.97540
	190	2.81134	2.91168	2.97530
In	180	2.73280	2.83876	2.91003
InL_{III} (Cu$K\alpha_1$)	189	2.73191	2.83802	2.90924
$B\varrho_{cal} = 221.923$	192	2.73289	2.83921	2.91042
	197	2.73472	2.84070	2.91208
Sn				
Ref. [5]				
Sb	183	2.56107	2.68047	2.77024
SbL_{III} (Cu$K\alpha_1$)	193	2.56048	2.67983	2.76964
$B\varrho_{cal} = 211.281$	195	2.56110	2.68032	2.77010
	195	2.56081	2.68021	2.76984
Te	188	2.46798	2.59523	2.69602
TeL_{III} (Cu$K\alpha_1$)	188	2.46804	2.59536	2.69617
$B\varrho_{cal} = 205.536$	194	2.46688	2.59405	2.69490
	194	2.46686	2.59403	2.69486

same run. The peak current values of these lines are listed in the third, fourth and fifth columns of Table 2. In Table 3 are listed the mean Bϱ-values of the L_{I} and L_{II} photo lines (column 2), the corresponding energy values (column 3) and the binding energies, including work function (column 4). Finally, column 5 shows the L_{I}, L_{II}, L_{III} and K binding energies relative to the Fermi level, i.e. with the work function $\phi_{s\mu}$ of the spectrometer material, 4.5 eV, subtracted [5]. The L_{III} energies, which are included for the sake of completeness, are those reported previously in ref. [5].

It is a difficult task to perform direct measurements with the photo electron method on the K level energies of the elements concerned in this investigation, using the present apparatus. X-ray absorption spectroscopy neither yields accurate values for the K energies. X-ray *emission* lines, on the other hand, can be measured with very high precision, and this makes possible accurate determinations of K level energies by addition of K emission line energies to L levels as obtained with the photo electron method [6, 7]. This procedure has been employed for the calculation of the K energies listed in the last column of Table 3, the K energies given being the mean of the $(L_{\mathrm{II}} + K\alpha_2)$ and $(L_{\mathrm{III}} + K\alpha_1)$ energies. The $K\alpha_1$ and $K\alpha_2$ energies are evaluated from the wavelength data in Table 1 with a conversion factor from XU to eV which is different from that used in Table 1, viz. the 1953 value given by Du Mond and Cohen [24]:

$$12\,372.2 \cdot \mathrm{keV\ XU}.$$

The reason for this is that all previous photo electron data are based on the 1953 least squares adjustment. This is the case not only for the above conversion factor, but also for the output data underlying the Bϱ–keV conversion tables employed [25]. Consequently, the K emission energy values involved in the K energies of Table 3 differ from those of Table 1 by approximately 0.5 eV. It may be mentioned that the development of the photo electron method has made possible a determination of the keV XU conversion factor by a method which was originally proposed by K. Siegbahn [26]. This determination is now being carried out at this institute.

3.2 Error estimate

Altogether 40 photo electron measurements have been made, and since all these measurements can be assumed to have the same relative uncertainty, the error assigned to one single measurement can be calculated from the standard deviation of the whole assembly of data [27]. The relative spread in momentum, expressed as the standard deviation, is found to be $3.5 \cdot 10^{-5}$. Twice the standard deviation of the mean of four measurements makes $3.5 \cdot 10^{-5}$, which is assigned as the error of the mean, corresponding to a confidence coefficient of 95 %. The error limits given in column 3 of Table 3 are twice as large, since this column lists the *energies* of the photo lines. The relative errors of the calibration lines are $3.5 \cdot 10^{-5}$ in terms of momentum according to ref. [5]. This error is included by quadratic addition in the error limits given in column 4 of Table 3. It should be pointed out that uncertainties in fundamental constants and X-ray data are not taken into account.

According to section 2.3 the error limits of the $K\alpha$ emission lines are ± 0.2 eV for Ag, Cd, In and ± 0.3 eV for Sn, Sb, Te. Quadratic addition of these uncertainties with those of the corresponding photo electron measurements yield K level error limits of ± 0.5 eV for all elements included in this investigation.

Table 3. Mean values of momenta and energies of photo electrons, binding energies.

Level	$B\varrho$ (gauss-cm)	E_{photo} (eV)	B.E. + ϕ_{sp} (eV)	B.E. (eV)
Ag L_{I}	219.945	4236.8 ± 0.3	3810.7 ± 0.5	3 806.2
L_{II}	227.191	4519.3 ± 0.3	3528.2 ± 0.5	3 523.7
L_{III}				3 350.8
K				25 513.6
Cd L_{I}	214.352	4024.9 ± 0.3	4022.6 ± 0.4	4 018.1
L_{II}	221.996	4315.9 ± 0.3	3731.6 ± 0.4	3 727.1
L_{III}				3 537.3
K				26 710.6
In L_{I}	208.399	3805.3 ± 0.3	4242.2 ± 0.4	4 237.7
L_{II}	216.489	4105.2 ± 0.3	3942.3 ± 0.4	3 937.8
L_{III}				3 730.0
K				27 939.4
Sn L_{I}				4 464.5
L_{II}				4 156.2
L_{III}				3 928.8
K				29 199.7
Sb L_{I}	195.336	3344.7 ± 0.2	4702.8 ± 0.4	4 698.3
L_{II}	204.436	3662.4 ± 0.3	4385.1 ± 0.4	4 380,6
L_{III}				4 132.2
K				30 490.9
Te L_{I}	188.147	3103.7 ± 0.2	4943.8 ± 0.4	4 939.3
L_{II}	197.849	3431.0 ± 0.2	4616.5 ± 0.4	4 612.0
L_{III}				4 341.2
K				31 813.0

4. Comparison of X-ray and photo electron data

The most surveyable way of comparing the energies of Table 3 with the corresponding X-ray absorption energies over a range of Z values is by means of modified Moseley diagrams. In Fig. 2 are shown such diagrams for the L_{I}, L_{II}, L_{III} and K levels. Here the photo electron measurements (filled circles) are plotted together with representative X-ray absorption data. The scale, indicated in the figure, is the same in all four diagrams. The elements Ag and Cd have s type valence electrons and, starting with 49In a new subshell of p symmetry is built up. The break in the Moseley lines at this point is clearly visible for the L_{III} level for which straight Moseley lines are to be expected [7]. For the curving lines representing the L_{I}, L_{II} and K levels, the existence of a break is more uncertain. The interpretation given to the experimental points in the diagrams is, however, quite reasonable. We have chosen to draw the L_{I} and L_{II} curves so that a constant separation (Hertz' law of screening doublets) is preserved.

It has been pointed out previously that discrepancies exist between the results obtained from X-ray absorption spectroscopy and those of the photo electron method which imply that the two methods do not measure identically the same quantity [3, 5, 6, 7]. The large deviations observed for the L_{I} level of the rare earths [7] is less pronounced for the elements of Fig. 2. However, in all four diagrams the Ag and Cd absorption data are higher than photo electric data, which may be correlated to the fact that these elements have s type valence electrons. Such elements should

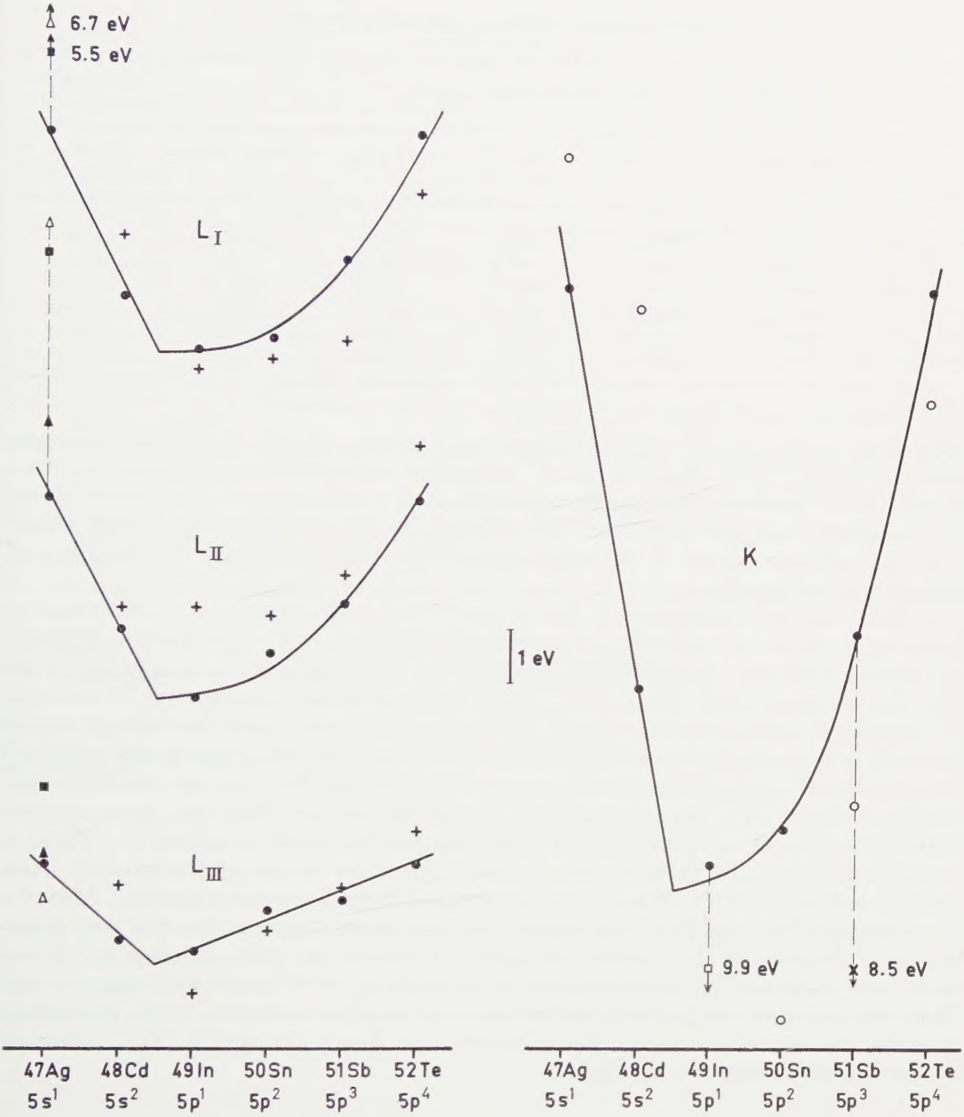

Fig. 2. Modified Moseley diagrams of the present measurements according to the last column in Table 3, together with representative X-ray absorption data taken from refs. [19, 29–34]. The scale, in electron volts, indicated in the figure, is the same in all four diagrams.

● Present
△ Coster and Mulder
+ Sandström
○ Ross
□ Leide
× N. Nilsson
■ van Dyke and Lindsay
▲ Nordfors

Table 4. Spin doublet separation in eV.

Element	X-ray emission spectra					Photo electron spectra
	$K\alpha_1$–$K\alpha_2$ (Table 1)	$L\beta_1$–$L\alpha_2$	$L\eta$–$L\,l$ (Ref. 22)	$L\gamma_5$–$L\beta_6$	Mean values	L_{II}–L_{III} (Table 3)
Ag 47	172.4	172.8	172.4	172.4	172.5	172.9
Cd 48	189.4	189.7	189.4	189.3	189.5	189.8
In 49	207.8	207.9	208.2	207.6	207.9	207.8
Sn 50	227.2	227.4	227.3	227.1	227.3	227.4
Sb 51	248.3	248.2	248.0	248.8	248.3	248.4
Te 52	270.7	270.8	270.3	270.5	270.5	270.8

differ from others in that the "valence-electron-configuration" excitation states are more pronounced [28]. Most of the K absorption data are those reported by Ross in 1933 [31]. They were obtained from measurements with a two-crystal spectrometer and ionization chamber detection. The critical absorption wavelengths were taken from the inflexion points of the transmission curves and not from the variation of absorption coefficient.

A check on the consistency of the present X-ray diffraction and photo electron measurements can be made from a comparison of the L_{II}–L_{III} spin doublet splitting as obtained with the two methods. This quantity is obtained as the separation of the $K\alpha_1$–$K\alpha_2$ emission lines and L_{II}–L_{III} photo electron lines, respectively. It can also be obtained from L emission spectra. According to Parratt [28], the atomic states involved in emission are pure "ionization" states and the same states are supposed to be produced in the photo electric process. This implies that the spin doublet separations obtained with the two methods should be identical and also gives support to the procedure of computing the K level energies described in section 3.1. There is a complete agreement between the crystal diffraction values of the present work listed in column 2 of Table 4 and those obtained from L emission spectra. Also, for the elements 49In to 52Te crystal diffraction and photo electron data fall well inside the limits of error. For the elements Ag and Cd, however, the photo electron and X-ray spectroscopy data differ systematically by as much as 0.5 eV and 0.4 eV respectively. These discrepancies are probably significant and may be correlated to the deviations for these two elements between photo electric and X-ray absorption data, witnessed by the Moseley diagrams in Fig. 2.

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